The Chemical Age

A Weekly Journal Devoted to Industrial and Engineering Chemistry

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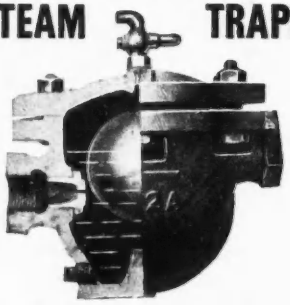

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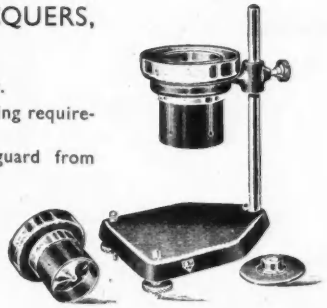

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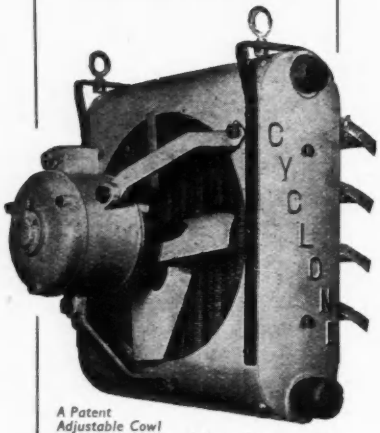

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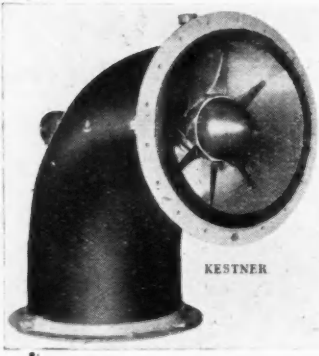

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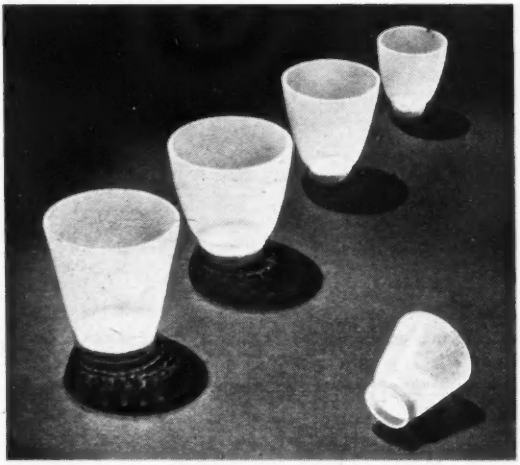

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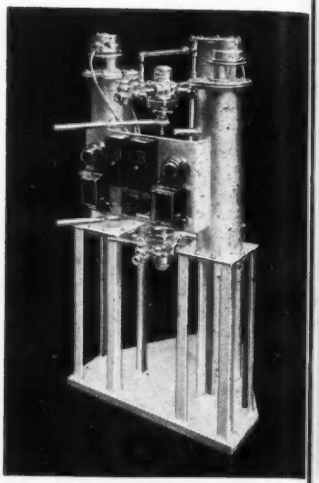

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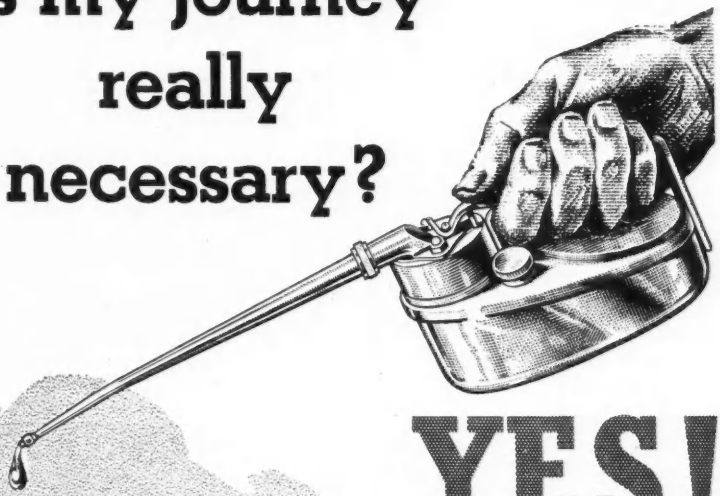

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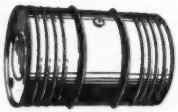
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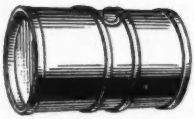
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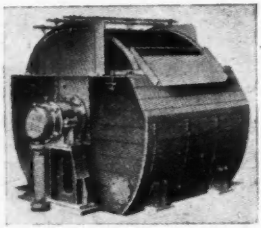

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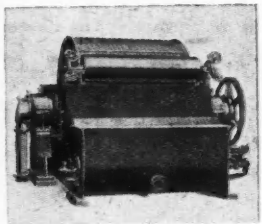

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More about Chemicals from Coal

THE leading article in THE CHEMICAL AGE of June 10 contained some thoughts on Mr. Bennett's views on the manufacture of chemicals from coal. A parallel but distinctly different view of the subject was given by Dr. J. G. King, the Director of the Gas Research Board, in his Stevens Memorial Lecture to the Junior Institution of Engineers on May 5. We have referred in these columns on many occasions to the desirability of the home refining of petroleum. If the petroleum industry decides to refine petroleum in this country a good many of the developments projected on the production of oil from coal will be unnecessary for the time being; they will become necessary only when the world supply of petroleum begins to fail—probably a generation or two hence. If in addition we can set up synthetic chemical industries based on the products of the refineries, the case against the replacement of oil by coal will be vastly strengthened.

Dr. King, however, considered what steps ought to be taken towards the better utilisation of coal for the production of oil and petrol. Carbonisation produces coke, tar, gas, and benzol, all of which can be used in one way or another for this purpose. Before the war a large proportion of the 2 million tons of tar made (720,000 tons) was used on the roads. The remainder was distilled to provide naphtha, creosote, and other products for chemical industry, leaving as a residue, pitch, for which the market fluctuated in an unsatisfactory way, and for which there was relatively little demand in this country. The contribution that tar made to the oil market was therefore little more than the benzol produced (105 million gallons). During the war the use of creosote pitch has made a considerable contribution to our requirements of oil, but after the war road tar will certainly be made again and most of the creosote may be hydrogenated. Some pitch and creosote may be available, however, and these can be disposed of at fuel oil prices in this way. If the 600,000 tons now being made yearly were continued, they would replace 500,000 tons of heavy fuel oil from petroleum.

From the chemical point of view the most interesting possibilities lie in hydrogenation. It is known that the hydrogenation process could produce three types of product according to the catalysts used. These catalysts may thus be divided into three types: (a) those which operate at high

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temperature (500°C.), have high cracking power, and give a rather lower yield of spirit rich in aromatic hydrocarbons; (b) those with a high saturating power and which operate at a low reaction temperature (400°C.); (c) those which operate at an intermediate temperature and which favour isomerisation and therefore increase of octane number. The economics of the process were not disclosed by Dr. King, but he points out that the coal consumption is such that the manufacture of 10 million tons of motor spirit would consume over 16 million tons of process coal and 35 million tons of coal altogether. Probably the principal use for the hydrogenation process is in the treatment of tar and tar oils for the production of chemicals or fuels. Results quoted by Dr. King show that all the tar made could be hydrogenated to spirit, if plant were available, with a loss of only 3 to 4 per cent. of untreatable residue. One ton of tar yields about 0.6 ton of spirit.

The Fischer-Tropsch process is, Dr King believes, most valuable as a source of chemicals. He points out that for a source of chemicals rather than of fuels, high thermal efficiency is not so essential, but the true value is not so low as the 25 per cent. usually quoted. The joining of a Fischer-Tropsch plant into a unit already producing gas and/or coke might well lead to an overall efficiency not realisable in an independent plant. Assuming both coke and gas available, and cracking some gas to raise the H_2/CO ratio of water gas (made from coke) to 2 : 1, it can be calculated that 3.45 tons of coal only are required to produce 1 ton of primary product. The calculation assumes synthesis in three stages, the spent gas after each stage being cracked with steam to make additional synthesis gas, and the utilisation of the heat of reaction in the form of steam to operate the entire plant, including the manufacture of catalyst. This figure of 3.45 tons is equivalent to a thermal efficiency of 42 per cent., which will not be improved until the yield of 125 gm. of primary product per cubic metre can be made to approach more closely the theoretical figure of about 180 gm. The thermal efficiencies of the hydrogenation and synthesis processes are therefore of the same order.

An important asset of the Fischer-Tropsch process is the fact that lubricating oil of high quality can be made from the primary product. The exact process has not been disclosed, but it involves thermal cracking of the middle oil, followed by the polymerisation of the olefinic products of cracking with aluminium chloride. Yields of the order of 35 per cent. by weight have been claimed, but it is possible that these may be appreciably higher if Fischer's claims are justified for the direct production of unsaturated oils by the use of an iron catalyst at 15-20 atm. pressure. A synthesis plant of quite moderate size could supply a fair proportion of the half-million tons of lubricating oil imported (1938).

Coal gas (town gas or coke-oven gas) contains about 2 per cent. of ethylene and 0.5 per cent. of propylene, which could be recovered if a liquefaction technique were developed on a large scale within the carbonising industries. The application of such a process in the manufacture of synthetic ammonia has proved successful over a long period and there seems no reason why it should not be applied to ethylene recovery. The 100,000 tons of ethylene (and propylene) would be an immense asset to chemical industry. The process could also be extended to allow of the removal of part of the methane. If one-third of the methane in town gas were liquefied in this way it could replace 1 million tons of gas oil or petrol.

During the war we have not been idle in working out, both on the small and on the large scale, methods for the production of methane, ethylene and other gases from coal gas. We shall not be surprised if the chemical industry finds itself presented soon after the war with a great opportunity for the synthetic production of chemicals on a large scale derived from coal primarily through carbonisation. It is not impossible that there may be a parallel opportunity from the home refining of oil. It is known, for example, that the Minister of Fuel has set up a Committee to enquire into the interrelation of raw materials from oil and coal. Is the chemical industry equipped to take advantage of these important developments? They will require large capital and a good deal of courage in setting forth into new manufacturing fields.

NOTES AND COMMENTS

Silver Jubilee

ALTHOUGH THE CHEMICAL AGE celebrates its twenty-fifth birthday this week—the first number was dated June 21, 1919—no fanfare of trumpets can be employed to mark the occasion. The journal, born of one war, reaches full maturity in the thick of another whose advent was little imagined when the first proofs were passed for press. It is amusing, however, and more than a little chastening, to look back to the original leading article, and to see to what extent we in this country have allowed things to slide during the intervening period. At that time, we read, there was "a general feeling that no sufficiently serious attempt has hitherto been made to meet the demands of the chemists of this country in the way and to the extent they have been provided for in America and in Germany." Though there are now a few stirrings perceptible, it would still not be especially unfair to print the same statement to-day. Further on it is remarked that "as a result of the war there has sprung up among industrial chemists some tendency to regard any serious opposition as improbable; but it must be remembered, in spite of the severity (*sic*) of the peace terms, that Germany as a nation possesses great powers of recuperation, and that in the near future she will certainly make strenuous efforts to regain her supremacy in chemical production." Considering that this regained supremacy all but cost us our lives and our country, it is hardly necessary to insist on the truth of that remark. The rise of American chemistry is likewise envisaged, and it is fortunate for us that we can regard this as an asset, both now and in the long run.

British Chemists

AN interesting feature of the first number of THE CHEMICAL AGE is the fact that contributions included an interview with the President of the Society of Chemical Industry of the time—Professor Henry Louis—and an article by next year's holder of the same office, Professor E. K. Rideal. Chemical engineering was represented by Mr. S. J. Tungay, this year's chairman of the Chemical Engineering Group of the Society of Chemical Industry. Of those eminent chemists and others who sent the journal their good wishes for a prosperous career, many are no longer with us, but the point of view expressed by practically all of them was that more recognition should be given to British chemists. At no time was the neglect of native talent more in evidence than in the years immediately preceding the last war—Dr. Charles Carpenter, of the South Metropolitan Gas Company, even went so far as to write his belief that "if the country had been offered in pre-war days 'cheap' energy from Germany through a submarine cable, the Government would have supported the proposal." Professor Louis, too, advocated the blowing of our own trumpets a little more loudly, citing the "Haber" process, the "Bunsen" burner, and the "Welsbach" light as instances of the way the Germans tacked their names on to applications they had made of inventions born elsewhere. Perhaps this time we have learnt our lesson. Twice we have proved (if proof be needed) that our chemists are second to none. Is it too much to hope that some practical acknowledgment of this fact may be part of our recognised policy—whether official or industrial—in the years ahead?

War Diaries

IN the first weeks of the war we strongly advised our readers to keep records of their war activities, adventures and, if unhappily these should arise, tribulations. Memories get quickly blurred, and anything like an accurate and detailed account of business firms' experiences can only be obtained by regular entries made at the time. The permanent value of such records has been strikingly proved in the case of the publishing house from which this journal appears. The chairman recently published in book form his *Fleet Street Blitzkrieg Diary* which has been widely claimed as a valuable documentary of the war. Air-raids are only a part of the exceptional experience through which British firms have passed during the last four-and-a-half years. It is of major importance that all charges and adaptations caused in industry by Government regulation and control should be on permanent record At this moment, when the Allies are approaching the final phase of a unique historical chapter, we

venture to renew our appeal to readers to furbish up their war diaries and see that they are kept up to date from now onwards. When the censorship is abolished and a free nation can once more get to grips with the planners and controllers, the task will be easier if an abundance of such material is readily available.

Chemicals for Plastics

IT is probably not altogether a coincidence that the chemical requirements of the plastics industry, in the way of raw materials, should have come to the fore as a subject for discussion at about the same time both in this country and in the United States. We are publishing in this issue a lecture on the subject by Dr. Worboys, of I.C.I., and in the March, 1944, number of *Chemical and Metallurgical Engineering* (p. 109), an article by Mr. J. W. Raynolds, deputy-director of the Chemicals Bureau, War Production Board, Washington, deals with very much the same subject. It would appear that the inter-relation between the chemical industry and the plastics industry has gone further in America than here. Mr. Raynolds speaks of "many new facilities" constructed or planned for the purpose of supplying the chemical requirements of the plastics industry, and states that his Bureau, having determined the country's requirements in terms of compounded products, such as plastics, breaks these down into their chemical components. "Each important chemical," he says, "has been studied in this manner to determine the supply requirements." This study, of course, includes also the plasticisers and organic solvents necessary for the commercial application of plastic products.

A Gigantic Demand

AMERICAN production of synthetic resins is at present more varied than ours, and Mr. Raynolds treats each of the different groups separately, starting with the two general classes: thermoplastic and thermosetting. The former class includes cellulose compounds, vinyl compounds, rubber derivatives, and a miscellaneous group (coumarin-indene and beta-pinene resins). It is not our purpose here to summarise Mr. Raynolds's article, but a few figures will give some idea of the increase that must have been necessary in the output of intermediates, such as acetic acid, acetic anhydride, acetylene, butyl alcohol, calcium carbide, caustic soda, and chlorine—to mention only a few of the most versatile. United States production of the lacquer and plastic grades of nitrocellulose, for example, has increased from 37,000,000 lb. in 1940 to 60,000,000 lb. in 1943; of the plastic grade of cellulose acetate, from 24,000,000 to 55,000,000 lb. in the same period. The ethyl cellulose group, including cellulose-acetate butyrate and propionate, has risen from 8,000,000 lb. in 1941 to 36,000,000 lb. (projected) in 1944, while the vinyl resin group has expanded from 72,000,000 lb. in 1941 to 178,000,000 lb. in 1943. Comparable increases might be quoted for the thermosetting types—phenol-formaldehyde, urea-formaldehyde, and alkyd resins—but enough has been said to show the vast demands made upon the chemical industry. In yet another direction there is the insatiable need for organic acids to produce plasticisers, for coal-tar solvents, ester solvents, and petroleum solvents. "All told," Mr. Raynolds concludes, "the future looks promising for the chemical and synthetic resin industries."

New Control Orders

Acetylene

ACETYLENE, when compressed in a cylinder in a homogeneous porous substance, with or without acetone or other solvent, is exempt from the provision of the Explosives Act, 1875, if certain conditions prescribed by Order No. 9 of the Home Secretary, dated June 23, 1919, are complied with. A new Order (S. R. & O. 1944, No. 667) exempts acetylene in cylinders manufactured outside the U.K., and either owned by H.M. Government or a Dominion or Allied Government, or intended for export, which do not comply with those conditions, provided that the conditions in the Schedule to the new Order are complied with.

The conditions in the new Schedule deal with the place and method of charging the cylinder, with marking and examination, with the amount of solvent added, and with the pressure to which the cylinder is charged—in no case to exceed 225 lb./sq. in. at 60°F.

A bequest of £1000 to the Physical Society to found a Boys prize or lecture to further interest in experimental physics is included in the will of the late Sir Charles Boys, LL.D., F.R.S.

Recent Developments in Analytical Chemistry—X

(From Our Analytical Correspondent)

THE appearance of the first number of the British Chemical and Physiological Abstracts C is a landmark of some note for analytical chemists. The abstracts are a very real contribution to the literature of analytical methods, and the bureau is to be congratulated on the thorough way in which it has inaugurated the new development.

Electrochemical Deposition

The determination of metals by deposition on an electrode and weighing has been extended in an interesting fashion by a method devised by Williams and Johnson.[1] The determination of pectin is normally carried out by some such method as conversion to calcium pectate. However, if a solution containing pectin or pectates is treated with an ion-exchange resin to remove material which would normally come under the heading of "ash," *i.e.*, metallic ions, the result is a colloidal solution containing negatively-charged colloids. These colloids can be deposited in a weighable form on a platinum anode by passage of a current. Working with a solution containing from 5-50 mgm. of pectate, and using pure mercury as the cathode, a current of from 5-20 milliamps. (220 volts) is passed for 5 hours. A compact deposit of pectin collects on the platinum-gauze anode. This is dehydrated by three minutes' immersion in absolute alcohol followed by a similar immersion in anhydrous ether, and the anode, covered now with a thin uniform film of colloid, is dried for half-an-hour at 105°C. The speed of analysis is greater than with the more usual methods of estimation, while the accuracy is as great.

Copper may be determined in cast iron by direct micro-analysis.[2] Variations in apparatus, conditions, and impurities present have been thoroughly investigated, and it is recommended that a slightly modified Clarke-Hermance cell[3] be used. The sample is dissolved in nitric acid, but it is not necessary to remove the acid before electrolysis, as the oxides of nitrogen formed in the course of the determination are removed by a stream or air which also acts as stirrer. With a current of 2.5-2.8 volts, deposition is complete in 25-30 minutes, the film of copper being free from iron, and without blisters. If molybdenum is present it is claimed that the addition of phosphoric acid or of ammonium bifluoride effectively prevents the deposition of this metal. Other authors[4] have dissolved the sample in sulphuric acid, and then treated the solution with ferric sulphate or with nitric acid to oxidise any ferrous iron. A current of 2.1-2.2 volts is passed for 45 minutes, and stirring is again by means of an air stream. An alundum thimble is inserted in the cell as a diaphragm. If the sample is either cast iron or alloy steel, this simple electrolysis is normally satisfactory, but it is recommended that where much molybdenum, tungsten or cobalt is present, the plated metal is best stripped from the electrode, and then determined colorimetrically as cuprammonium ion.

Copper in lead and tin base alloys has also been estimated electrolytically with success.[5] The major part of the copper is taken out by treatment with nitric acid, tin (and antimony) being removed from the residue by heating with ammonium iodide. The copper-lead portions are then combined, lead is precipitated as sulphate, and copper is deposited electrolytically. Alternatively, the principles of internal electrolysis may be applied to these alloys,[6] copper and lead being deposited on iron and cadmium electrodes in a Collin-Sand apparatus.

Determining Copper in Ore

Copper-bearing ores, which may contain considerable amounts of extraneous metals, often present a problem if it is desired to estimate the metal electrochemically. Potassium ethyl dithiocarbonate, $C_2H_5.O.CS.SK$—long known as a qualitative reagent for a wide range of metals,[7] and better known as potassium xanthate—has (actually in the form of the sodium salt) been put forward as an aid in this connection.[8] This reagent should not be confused with sodium diethyl dithiocarbamate, $N(C_2H_5)_2.CS_2Na$, also a well-known reagent for copper.[9] Properly employed, the xanthate separates the copper from the ore in a form pure enough for electrolytic analysis. Half a gram of ore is decomposed by any recognised procedure, and is then fumed with sulphuric acid. It is cooled, and 75 ml. of water added, together with several gms. of tartaric acid. The whole is boiled, and lead sulphate and other insoluble matter removed by filtration. The solution is made strongly alkaline with ammonia, and then cooled; 25 ml. of a fresh 10 per cent. sodium xanthate solution is added, and the whole is stirred till the yellow copper xanthate coagulates and the supernatant liquid clears; a sharper separation results if the solution is thoroughly cooled during this process. The precipitate is

filtered off, washed several times with water, and ignited to burn off the filter paper, and to remove traces of arsenic and selenium. The xanthate is then dissolved in nitric acid and electrolysed. If silver is present it will also precipitate, and should be removed by a chloride precipitation before adding the sodium xanthate. Cobalt and nickel may be estimated in the electrolyte, after deposition of the copper, by such normal methods as the dimethylglyoxime precipitation of nickel. The error for copper is small—0.1 per cent.—and may be even smaller if the copper is estimated iodometrically, which is also feasible.

Polarography of Copper

The polarograph has been applied to the determination of copper in brass plating on iron or steel.[10] Ammoniacal ammonium persulphate is used to dissolve the brass plate, while the supporting electrolyte consists of ammoniacal ammonium chloride and ammonium sulphite, gelatine being added as a maximum suppressor. After obtaining the polarogram, it is only necessary to determine the relative heights of the copper and zinc waves—not the *absolute* amounts. Consequently, elaborate control of such factors as temperature, dropping rate, etc., is not called for.

Traces of copper, lead, and cadmium have been shown to cause corrosion in high-purity zinc alloys.[11] and it is therefore important to be able to determine how much of these metals is present. The polarograph has again proved suitable for this purpose, giving an accuracy of $\pm 1 \times 10^{-4}$ per cent. of the sample weight. Oxygen is removed prior to the analysis by passing nitrogen for 15 to 20 minutes. Copper may also be determined polarographically in a wide range of materials if alkaline sodium potassium tartrate is used as the supporting electrolyte.[12] Fuchsin is added as a maximum suppressor. Oxygen is first removed. Such an estimation can be carried out on iron pyrites, and on aluminium or white metal alloys, without prior separations.

A systematic examination of the possibilities of polarographic analysis on unknown materials has been commenced by Linganes.[13] Complete data have been collected for eight metals, including copper, lead, zinc, cadmium and tin, in nine different supporting electrolytes. By using these data, the necessity for first calibrating the apparatus with standard solutions of the substances to be determined is avoided. A useful chart of half-wave potentials of the metals is given. Copper has also been included in another polarographic survey.[14] this time concerned with the nature of base electrolytes. Aqueous ammonium acetate gives satisfactory waves, free in general from maxima, with copper, nickel, lead, cadmium, manganese, cobalt, and zinc. Preliminary work has shown that most of these metals, and some others can also be polarographed successfully in solutions containing ethanolamines, together with some other substances, which serve to shift and separate the waves of metals which normally occur together.

Other Methods for Copper

Dithizone has been recommended for the rapid spectrophotometric estimation of copper.[15] Where the *p*H of a solution is less than 3, the dithizonates of copper, silver, gold, mercury, bismuth, and stannous tin are extracted by chloroform or carbon tetrachloride, while practically all other metals are unaffected. When this extract is shaken with acidic potassium iodide, all the complexes except that with copper are destroyed. If the transmittancy of the solution is then determined, the amount of copper may be read off from a calibration curve. The method has been applied to the determination of copper in foods, steel and solder, and has an accuracy of ± 0.3 g.

Copper may be precipitated by anthranilic acid, and an analogous method has been designed for its precipitation by 8-quinolinecarboxylic acid.[16] The copper salt, dried at 120° C., has the formula $(C_{10}H_6NO_2)_2Cu$, and the precipitation is carried out with a saturated solution of the reagent in neutral or weakly acid boiling solution. The method appears to be useful in the presence of lead, nickel, mercury, cobalt, cadmium, and zinc, but silver and gold also precipitate under these conditions.

Using 8-hydroxyquinoline, it has been found preferable to precipitate for a gravimetric estimation in the presence of alkaline sodium tartrate.[17] If acetic acid solution is used results tend to be too high. As an alternative, the 8-hydroxyquinoline complex can be rendered suitable for colorimetric determination.[18] Copper is completely extracted by shaking with a chloroform solution of 8-hydroxyquinoline anywhere in the *p*H range 2.7 to 7.0. If the extraction is maintained at *p*H 4.0, it is possible to estimate the copper thus extracted in the presence of forty times as much cadmium or zinc, using the transmittancy at 410 mμ.

The behaviour of 1 : 10-phenanthroline and its derivatives with copper, iron and molybdenum, has been investigated,[19] and a method devised for the estimation of copper with 1 : 10-phenanthroline itself.[20] An ammoniacal solution of the copper salt is treated with the reagent, and then reduced with hydroxylamine, giving a brown-orange, strongly-coloured cuprous compound. The colour may be rendered stable for twenty-four hours, and it is best to determine the transmittancy at 435 mμ. Cadmium, cobalt, nickel, and zinc should not be present, while certain anions also interfere.

Sodium diethyl dithiocarbamate has been employed for the rapid determination of copper in ferrous materials, photometrically.[21] Nickel must first be removed by dimethylglyoxime, but it is possible to carry out a complete determination on a nickel-containing steel in 20 minutes after solution of the metal has been achieved. The method is suitable for routine work.

Spectrography

Copper and copper-rich alloys have been analysed by means of the arc spectrum.[22] The first problem was to ensure constancy of the source, and it was found impossible to do this solely by control of the electrical conditions. Success was achieved by allowing the light from the arc to fall on a photocell connected to a microammeter, and adjusting so that the *total* visible light from the arc was maintained at a constant value. By this means satisfactory results were obtained from a wide variety of copper-rich alloys. Since it was found that the copper volatilised more readily from the cathode (by an oxidation process) the practice was to arrange the sample as cathode, in conjunction with a pure copper anode. In this way it was shown that there was no danger of selective distillation of the impurities.

A number of recent publications have dealt with what has been termed a "multi-source unit" for spectrographic analysis.[23] The apparatus is devised to cover the widest possible range of conditions, suiting any emission spectrographic analysis. Since it may at one time be preferable to use a d.c. arc, at another an a.c. condensed spark, the arrangement has been not only to provide these, but also to produce an almost continuous grading from the extreme of arc conditions to the extreme of spark conditions. The precise conditions anywhere in this range which have proved suitable for a particular estimation can thus be accurately reproduced. With the multi-source unit various alloys of copper, aluminium, iron, magnesium, and zinc have been investigated, while with a similarly devised unit other authors[24] have made determinations of zinc in brass.

The application of infra-red spectrography to organic analysis is becoming widespread. Infra-red spectrometers have been described by Wright,[25] and by White and Heigh.[26] The latter instrument is of the usual Littrow type, with a 5 cm. rock salt prism, and an absorption cell equipped with a precision manometer, suitable for handling gas samples, permanently built into the set-up. Simple hydrocarbon mixtures may be analysed with an accuracy of better than 1 per cent. of the sample. Nielsen and Smith[27] have devised a method suitable for the analysis of binary and ternary mixtures of nitroparaffins. Beer's Law must hold, and a set of wavelengths is chosen whereby one of the components has strong absorption, while the others have weak absorption. In the ideal case, of course, one would have strong absorption while that of the others would have a negligible value, but although this cannot in general be attained, the method is nevertheless serviceable. Determinations of 2-nitro propane and nitromethane in nitroethane, and of 1-nitropropane and nitroethane in 2-nitropropane, are representative of analyses which this method makes possible.

A similar principle is carried to a specialised application in the analysis of butadiene,[28] where a single wavelength is chosen such that butadiene is more transparent than the impurities associated with it, and at the same time the absorption coefficients of the impurities are of the same order of magnitude. Butadiene at a regulated pressure is admitted to an absorption cell, and the value for butadiene concentrations from 90-100 per cent. can then be determined in duplicate in six minutes.

Infra-red spectroscopy as a rapid and efficient means of qualitative analysis (since the sample is conserved) both for organic and for specific compounds, has been recommended by Stafford and Barnes.[29] Extension of the method to the analysis of multi-component systems, such as the estimation of *cyclo*-hexanone in *cyclo*-hexane, and of butyl alcohol and dibutyl ether in butyl acrylate, has also been described.[30] Finally, infra-red spectrophotometry of mixtures in which Beer's Law does not necessarily hold has been achieved,[31] and analysis by this means of a complicated mixture such as a six-component system of C_4 hydrocarbons is recommended as being frequently the only method of analysis practicable.

The Mass Spectrometer

A completely new instrumental method of analysis (in the chemical sense) has now been developed in the form of the mass spectrometer.[32] The mass spectrograph is in general an instrument for sorting molecules, by giving them an electric charge and then subjecting them to heterogeneous magnetic field while in motion. As a consequence the molecules of different masses follow different paths, while those of the same mass will follow identical paths. Recorded photographically or by an amplifier and recording oscillator, indications are given of the mass—or, more precisely, of the mass and charge—of the different molecules. The mass spectra for different molecules will be characteristic, and since they may be regarded as additive, it is therefore possible, from a knowledge of the spectra of the components of a mixture, to determine which of these is present, and in what proportions. Mixtures of paraffins and olefines, of benzene, toluene and xylene, and

of *n*- and *iso*-butane are representative of those which can be analysed. Only a small amount of the gas is necessary for the analysis, the time taken up is short, and the calculations are relatively simple. The method has proved especially applicable to analysis of the gases used in the preparation of butadiene, styrene and high-octane petrol. The sensitivity is so great (it will be remembered that several rare isotopes have been detected or confirmed by the mass spectrograph) that one part of oxygen in 10,000 parts of nitrogen can be detected.

Rubber Analysis

Infra-red spectroscopic methods, which have already been dealt with above in some detail, also find a use in the analysis of rubber stock compounded from natural and synthetic rubbers.[33] A simple procedure separates the rubber hydrocarbon from the various compounding ingredients. Infra-red examination then enables both the types of rubber present and the amounts of these to be determined.

A scheme of analysis for synthetic rubbers, with or without admixture of natural rubber, has been proposed by Stern.[34] The smell of a raw rubber, particularly on burning, is first noted, and the elements sulphur, chlorine and nitrogen are tested for by the usual Lassaigne's test. This alone may serve to type the rubber if it is a simple sample, and further specification is obtained by estimating these elements, while yet other determinations, such as the iodine value,[35] may prove useful. A modified colour reaction, carried out by treatment of 0.05 gm. of the sample with 0.05 ml. of bromine, warming for 30 seconds, adding 1 gm. of phenol and again warming, is described, enabling the final and certain identification of natural rubber to be made.

If the rubber is vulcanised, the tests so far described should first be carried out, but the presence of certain elements—*e.g.*, sulphur—will now not convey so much positive information. While the colour test is equally suitable for vulcanised rubbers, additional tests, such as the mode of disintegration in nitric acid and the swelling in various solvents, are also necessary. The exact amount of rubber in the vulcanised material is probably best carried out by a difference determination, estimating the various added ingredients first. The nature of the accelerator is difficult to decide, and a general knowledge of what is likely to have been used is essential if the determination is not to cause more trouble than it is worth.

The estimation of total sulphur in rubber is normally carried out by conversion to sulphate which is then weighed. However, the treatment takes time, and it has been found more rapid[36] to reduce the sulphate to sulphide by means of hydrogen iodide, distil off the sulphuretted hydrogen, and titrate this iodometrically. The method has many advantages—rapidity and accuracy being among the chief—but it has the drawback that it becomes difficult to handle when more than 5 mgm. of sulphur are present. In order to distinguish between total and combined sulphur,[37] total sulphur may first be estimated by combustion and absorption of the gases in alkaline hypobromite, giving sulphate. A sample is then extracted with methyl ethyl ketone, and a further sulphur estimation gives extractable sulphur by difference.

Where it is necessary to determine mercury in rubber which also contains iodine, the sample is decomposed[38] with sulphuric acid, iodine is expelled by boiling with nitric acid under controlled conditions which prevent loss of mercury, and the mercury is then determined by a Volhard with ammonium thiocyanate.

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Raw Materials for Plastics

Future Developments of the Industry

SOME interesting facts regarding the raw-material needs of the plastics industry were given by Dr. W. J. Worboys of I.C.I. in an address to the Institute of the Plastics Industry London Section, on May 25. These needs, he said, would be conditioned by the varied nature of the industry. It is worth while to compare his remarks with an article by Mr. J. W. Raynolds, deputy-director of the Chemicals Bureau, War Production Board, Washington, in *Chem. Met. Eng.*, March, 1944, pp. 109-113.

After some preliminary observations on the widely divergent processes covered by the plastics industry, Dr. Worboys examined the question of the major plastic materials now manufactured in the United Kingdom and the raw materials commonly used. "For the present range of thermosetting plastics," he said, "*i.e.* phenolic and amino moulding powders and resins, the main raw materials are the tar acids, phenol and cresol, together with urea and formaldehyde. For the cellulose group the main raw materials are cellulose, together with nitric acid for celluloid and acetic acid for cellulose acetate. For the acrylic resins we use acetone, cyanide, and methanol. Polythene and styrene require ethylene, at present derived from alcohol. The vinyl resins, *i.e.* the family which includes polyvinyl chloride, polyvinyl acetate, polyvinyl alcohol and interpolymers of these materials, are made from acetylene derived from carbide. Nylon, one of the newest of our plastic materials, is made from benzene or phenol. These are our main current raw materials, and I have purposely omitted reference to essential minor materials, *e.g.* camphor and other plasticisers."

Raw Material Groups

These raw materials could be grouped as follows:

1. Phenol, cresol, and benzene are coal-tar distillation products. Phenol, of course, is also synthesised from benzene.

2. Methanol, formaldehyde, urea, and nitric acid are products of the synthetic nitrogen industry, which itself is based on coal.

In other words, these materials rested on two domestic industries, tar distillation and synthetic nitrogen, which were well founded. Such raw materials should, therefore, be available in the quantities required, and at prices which would enable Britain to compete anywhere in the world.

3. Calcium carbide was the source of the acetylene required for the vinyl group, and carbide required for its manufacture coal, limestone, and cheap power. Before the war British requirements were imported from Scandinavia and Canada, countries with cheap water power. Two plants based on power generated from coal were now manufacturing carbide here, from which vinyl resins were obtained. The post-war economics of home-produced carbide could not be discussed now, but the importance of coal prices had to be appreciated. Should acetylene from domestic carbide not be an economic route for vinyl resins, recourse would then have to be made to imported carbide, or the possibility of making vinyl resins from ethylene would need to be examined. This process is now well established in North America, and a pilot plant has been operated in this country.

4. Alcohol, made from molasses, is the source of the ethylene required for polythene and styrene. Alcohol is also the raw material from which domestically-produced acetic acid is made, and acetone is manufactured from either alcohol or molasses.

Alcohol is made by the fermentation of molasses, but if adequate supplies of molasses were not available at economic prices, Dr. Worboys explained, that alcohol could be made by the fermentation of potatoes or corn or other waste agricultural products. If for any reason alcohol was not available, then ethylene could be obtained from coal or coke-oven gas or from the gases obtained from cracking oil. The concentration of ethylene in coal or coke-oven gas was generally low, and if Great Britain wished to recover ethylene from such sources, not inconsiderable technological problems of concentration and separation would have to be solved, if the resulting ethylene was to be sufficiently cheap. Similarly, the amount of ethylene in oil-cracking gases was not high unless the oil was cracked specially to produce ethylene. Oil-cracking gases generally contained propylene as well as ethylene, and from this propylene acetone could be made. Acetic acid was manufactured from coal in America by a process based on the interaction of carbon monoxide and methanol, a process which was quite possible here.

American Developments

Dr. Worboys then considered the plastic products now made in America but not in this country—or only in small quantities up to now. Examples were polyvinylidene chloride, polyvinyl butyral, ethyl cellulose, and cellulose acetobutyrate, and these required for their manufacture similar raw materials to those already used in Britain, namely, ethylene, acetylene, and acetic acid. The butyrals and butyrates, in addition, required butyl alcohol. Normal butyl alcohol was manufactured in Britain before the war by the fermentation of molasses, and *iso*butyl alcohol was now manufactured here from coal by a modification of the methanol synthesis. Newer plastic materials were appearing on the American market such as melamine formaldehyde, C.R.39, and other cross-linking resins; America also enjoyed a wider range of plasti-

cisers. Many of these were based on less common organic chemicals derived from the oil industry, and there was no doubt that the presence in the United States of a large petroleum industry did not produce many organic materials which at present were comparatively scarce here.

It should not be assumed that Britain was necessarily excluded from many new developments in organic chemistry because we had no oil industry. There was often more than one route to the preparation of a given organic chemical and, until recently, Britain had neglected her coal industry as a source of organic chemicals. Really active work in this field might produce surprising results.

"Here it should be remembered," Dr. Worboys said, "that a large proportion of the synthetic rubber made to-day is not based on oil. Even in the United States I believe that a substantial proportion of the synthetic rubber production is made from ethylene derived from alcohol rather than from petroleum gases. In Russia, too, where large quantities of petroleum are also available, synthetic rubber has been produced from alcohol made from potatoes or corn, and in Germany they produce synthetic rubber from coal and have succeeded in building up a large organic chemical industry without petroleum."

Thoughts for the Future

The conclusion drawn from this necessarily brief consideration of the raw material position was that so far as the synthesis of new polymers and interpolymers was concerned, development in this country should not be inhibited by any lack of suitable raw materials, but he urged that serious consideration should be given to coal as a source of organic chemicals. Without more knowledge of their eventual sources, it was not yet possible to say whether the raw materials required for the development of new plastic products would be available at prices enabling Britain to compete effectively in world markets. Production costs would vary with the scale of operation as well as with the chemical route followed, but there were additional points relating to economic production.

First, in this industry the raw material frequently represents only a small percentage of the total cost of the article finally sold to the public. Secondly, there was increasing evidence to show that the purity of the raw material often had a profound effect on the course of polymerisation reactions, and consequently, on the properties of the resulting polymer. In certain cases this might mean that the cheapest source of a raw material was not always the best.

Dr. Worboys hesitated to prophesy what new plastic materials were likely to appear in the next few years, but as an example, he predicted that there would certainly be new fibre-forming polymers developed. From these polymers, fibres would be spun which, while they would certainly find some normal textile applications, would because of their specialised properties also find specialised industrial applications. Further he said that from existing plastic materials or modifications of them, and from quite new materials, new wrought forms with a variety of desirable properties would be developed. The plastics industry should make a substantial contribution towards this country's post-war export trade. It was a new industry in all countries and it should be able to produce new products for export or enable old products to be presented more attractively. This was important because of the inevitable changes in the export trade that would flow from the industrialisation which had taken place in overseas markets during the war.

Canadian Industries, Ltd.

Annual Report

THE importance of the undertakings of Defence Industries, Ltd., in the construction, equipment, and operation of Government-owned plants for the manufacture of chemicals, explosives, and small-arms ammunition, as well as shell- and fuse-filling, is emphasised in the annual report of Canadian Industries, Ltd. Total costs incurred in these projects undertaken on behalf of the Canadian and United Kingdom Governments have so far exceeded $600,000,000. The income of Defence Industries, Ltd., is derived from management fees and from operations for which capital was furnished by the parent company; the income from this latter source was substantially eliminated in 1943 by the effect of the lower rate of production and the termination of some contracts. Virtually the entire income for 1943 was again declared in dividends, which totalled $382,500, in comparison with $430,000 in the preceding year. Net income from management fees received by Defence Industries, Ltd., from the commencement of the war to the end of 1943 represented less than one-fifth of one per cent. of all the costs incurred by the company on behalf of the two governments.

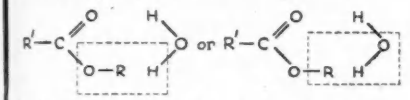
Funds for the construction and operation of new plants managed by Defence Industries, Ltd., were supplied by the Department of Munitions and Supply on behalf of the British and Canadian Governments. Canadian Industries, Ltd., originally invested $2,000,000 in the subsidiary company to provide it with funds to expand and operate two small plants taken over from the parent company. The company erected 10 large plants and 6 smaller works for the production of war-time chemicals and explosives, etc. For the first time since 1940 no major project was under construction at the end of the year. From a peak of 32,300 employees in March, 1943, there was a decline in succeeding months, but the average employment for the year was at approximately the same level as in previous year.

Modern Trends in Fat-Splitting

Development of Continuous Processes

by M. K. SCHWITZER

FATS and oils as encountered in nature are esters of glycerine and the fatty acids. The resolution of the glycerides into their components is one of the oldest branches of chemical technology and is known as "fat-splitting" or "saponification." Chemically, fat-splitting is the hydrolysis of an ester of the pattern:



Evidence supports the first type of reaction[1].

Until about 1932 it was generally held that aqueous hydrolysis of fats by caustic alkali solutions of water alone was a heterogeneous or two-phase process, that action took place exclusively at the oil-water interface, and that therefore it proceeded the more rapidly the greater the degree of dispersion of the fat globules throughout the aqueous phase. In 1932, Lester Smith demonstrated that in the saponification of oils with alkali, hydrolysis suddenly increases at a certain point, but remains substantially constant and relatively rapid until the process is approaching completion. This sudden increase in rate (with coconut oil at ordinary temperatures the acceleration is two-hundredfold) is due to the change from the initial sluggish saponification in the interfacial phase to the rapid reaction in the homogeneous solution. It is therefore, highly desirable that saponification should take place in one phase. This is in accordance with the theory that all hydrolytic reactions are homogeneous, proceeding in a single phase and not at the interface.

The older view, therefore, that a high degree of emulsification is required to increase the velocity of reaction, is to-day superseded by the desirability for making hydrolysis a one-phase process. At room temperature and atmospheric pressure neutral fats and oils dissolve only about 0.1 per cent. water. At higher temperatures solubility increases rapidly, and from Fig. 1 it will be seen that at 470° F., for instance, a fat of the tallow type will dissolve approx. 11 per cent. water and a fat of the coconut type approx. 23 per cent. water.[2] It is therefore understandable that when a homogeneous solution is required, high temperatures, and corresponding pressures to keep the water liquid, are desirable.

Hydrolysis is a simultaneous reaction of the reversible type. Two reactions proceed at the same time in opposite directions, each at its own specific rate. The net velocity of reaction is given by the difference of the velocities of forward and reverse reactions. Eventually a state of equilibrium is attained. Hydrolysis is therefore not carried to completion, but leaves a certain proportion of neutral oil. It has already been stated that high temperatures are desirable for fat-splitting in the homogeneous phase. How does the high temperature effect the rate of reaction and the equilibrium point? A study of the van't Hoff equation

$$\frac{d \ln k}{dt} = \frac{\triangle H}{RT^2}$$

for the change of the equilibrium constant shows that if hydrolytic reactions are accompanied by an evolution of heat, they will proceed farther at lower temperatures. In this equation k is the equilibrium constant, $\triangle H$ is the heat absorbed, T absolute temperature, and R the gas-law constant. Since saponification is a slightly exothermic process, the equilibrium point will be affected adversely with rising temperature, and will shift towards a lower yield of glycerine.

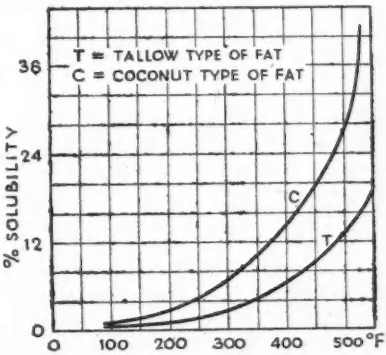


Fig. 1. Graphs showing solubility of water in fats at various temperatures.

On the other hand, the rate of homogeneous reactions increases rapidly with rising temperature. Generally for homogeneous reactions the rate increases two or three times for a 10° C. rise. The relative effect of an increase of temperature diminishes as the temperature is raised. In commercial processes the necessity of completing the operation in minimum time led to the use of the highest temperatures practicable in order to take advantage of the greater speed of reaction, regardless of a possibly unfavourable shift of the equilibrium point. It was thus possible to cut the operation time from 12-8 hours down to 2-½ minutes—about a thousandfold increase of reaction rate!

Catalysts are also used to increase the reaction

rate without resorting to high temperatures. Catalysts, however, have the disadvantages that they are expensive, that continuous operation is made rather difficult, and that their presence or their products of decomposition in the sweet water and fatty acids are undesirable impurities. In this connection it is interesting to note that the products of hydrolysis have an auto-catalytic effect on the reaction itself.

Modern processes obtain high yields of glycerine despite the tendency of the equilibrium point to shift in an unfavourable direction. This is achieved by working with the right proportions of water to fatty oil and operating in such a way that the products of hydrolysis are removed, thereby putting the system out of balance and causing the equilibrium point to shift in the required direction. This can be done, for instance, by obtaining a 60-70 per cent. glycerine yield in a first stage, and a 98-99 per cent. yield in a second stage.

The Older Processses

Before dealing with new processes a short review of the older ones, which are still responsible for the largest quantity of soap and glycerine production, will be of interest, and the relative merits and demerits of these will be more easily appreciated.

The oldest and simplest way of saponifying glycerides is by boiling them in open pans with caustic soda—though caustic potash is also used. Probably more than half the fat hydrolysed in this country and elsewhere is directly converted into soap by this method. Open soap-pans up to 200 tons total capacity are used. The fat charge is run into the pan in the liquid stage, and a caustic soda solution, or a strong lye from a previous boil, is added. Heating is applied by closed coils and live steam, the latter gently agitating the charge. To throw out of solution any soap which may have been dissolved in the water, brine is added in measured quantities. Saponification is complete in 12-24 hours and glycerine water, "soap lye," or "strong lye" is separated from the soap stock by settling. A similar process uses milk of lime as the hydrolytic agent. Saponification is carried out in an open soap-pan and the sweet water has a much higher glycerine concentration than ordinary soap lyes; also the colour of the soap and fatty acids is as good as that obtained in the soap-pan and usually superior to that from the Twitchell or autoclave splitting. About 50 per cent. excess of the alkali is necessary for conversion of the fat to lime soap. The rate of saponification should be kept low so as to give a coarse and porous calcium soap from which the glycerol can be easily washed out. After about 24 hours, saponification is complete and the lime soap separates as an almost solid curd, containing water and glycerine.

In the Krebitz method the curd is ground to a friable powder, and then washed with the smallest quantity of water requisite to remove the glycerine. The residual lime soap is said to contain 0.4 per cent. glycerol, while sweet waters with up to 90 per cent. glycerine content have been produced. This process is therefore suitable for small soap-makers who wish to send their sweet waters away for glycerine recovery. The Krebitz process, however, has found only limited application in English industry.

A method fairly extensively used in America and Germany, but which has not made great headway in England is the Twitchell method. Twitchell patented a reagent, which he prepared by acting on a solution of oleic acid in benzene, or preferably naphthalene, with sulphuric acid. The product appears to be of the general type $CO_2H(CH_2)_{17}(C_{10}H_6).SO_3H$. Similar reagents, claiming various advantages over the original Twitchell formula, came on the market, such as the Petroff and Pfeilring reagents, the Idrapid saponifier, Nekal B.X., etc. When the reagent is added to the aqueous fat it acts as a catalyst, promoting hydrolysis and converting the esters into glycerine and free fatty acids. The advantages of the process are its cheapness in steam consumption and a fairly high glycerine concentration of the sweet water, approaching the 15 per cent. of the autoclave process described below. A disadvantage is the somewhat lengthy period of the reaction, and a tendency to form dark-coloured fatty acids. The agent is employed in a concentration of about ½-1 per cent. of the fat present. When added to the aqueous mixture of fats it orientates its molecules so that the aliphatic part of the chain is directed towards the fat, and the acidic radicals, especially the sulphonic group, towards the water. It thus forms part of the same system and, in the presence of the strongly catalysing sulphonic group, hydrolysis is effected.

Sulphuric Acid Method

Saponification by strong sulphuric acid has also long been technically employed. The process depends on the partly hydrolytic, partly sulphonating action of strong sulphuric acid of which 5-8 per cent. is used. The charge is mixed mechanically and/or by live steam until hydrolysis is complete. That stage may be reached after 8-12 hours. The temperature is maintained throughout at 110-120° C. The fatty acids are extremely dark in colour and are cleansed by boiling with water after they are distilled. Superheated steam is passed into the fatty acids and the product condensed and collected. This process is not so efficient as the others mentioned above. Glycerine losses are high, and the fats are of low commercial value. The process is mainly applicable to acids which will require distillation in any case to purify them.

A process that might be rightly regarded as a step in the development from the various older saponification processes to modern continuous fat-splitting practice, uses pressures of about 100-200 lb./sq. in. The operation is usually conducted in a vertical, or sometimes horizontal, autoclave in copper or stainless steel, either jacketed or fitted with an internal coil, and

sometimes equipped with a mechanical stirrer. The vessel is charged with an oil and water mixture to about three-quarters its height, and about 3 per cent. of lime or zinc oxide or another catalyst is added. Steam is then admitted to the autoclave for 8-10 hours, depending on the yield of glycerine required (98-99 per cent. conversion of the fat to the fatty acids requires 10-12 hours). The products are free fatty acids, which can be used for soap-making, and a sweet water containing 12-16 per cent. glycerine.

Disadvantages

All the processes described have in common the following disadvantages: (1) fat-splitting takes from 8-24 hours; (2) the operation is discontinuous; (3) alkali, acid, or a catalyst is used to promote reaction; (4) the concentration of sweet water is comparatively low; and (5) the products are not always of the required quality owing to the presence of impurities and the reagents, or their products of decomposition.

The first two objections have a decisive influence on the dimensions of the plant, and therefore on the size of the buildings required. The long reaction time and the discontinuous nature of the operation require large vessels and considerable floor space, leading to greater heat losses as compared with the relatively small equipment of a modern continuous plant for the same annual output. Capital expenditure on plant for some intermittent processes is, however, less than for some continuous process plant. The use of chemicals or reagents has not only the disadvantage of higher production costs, but filter presses for the treatment of the sweet water and evaporators with salt boxes (in the soap-boiling process) are required. Filter-press operations are necessarily discontinuous and are often a cause of trouble. Most of the continuous processes produce free fatty acids, and for soap-making the caustic soda can thus be replaced by the cheaper soda ash. Objection number four has a considerable influence on the price of glycerine; sweet waters contain 5-15 per cent. glycerine against 30-40 per cent. in continuous processes, and evaporation of water is therefore more expensive.

In recent years chemical engineers have endeavoured to substitute continuous processes for batch operations wherever possible. The main advantages to be expected from a continuous process are: (1) smaller equipment required for the same output; (2) more accurate and automatic control; and (3) a product of homogeneous characteristics, only rarely obtainable to the same extent by batch operation.

Alongside the effort to make the process a continuous one, research was directed towards a method which would not employ any hydrolysing, emulsyfing, or catalysing agent, though there are some processes producing soap directly, instead of fatty acids, which employ chemicals. In most processes, however, water only is used for hydrolysis and no filtering of the sweet water is generally required, thus cutting out a troublesome step in glycerine production. As already indicated, the high concentration of the sweet water represents a considerable saving factor in the steam consumption of the evaporators.

The disadvantages of the continuous processes are that they hardly pay for small installations and that working under high temperature and pressures increases the risk of blow-outs and accidents. High pressure boilers and oil or Dowtherm heating equipment are also required, and, if soaps are to be produced, the fatty acids obtained by most of the continuous methods require more equipment for carrying out neutralisation. It is therefore understandable that the small-scale soap-boiler "cannot be bothered" with the multiplicity of fairly complicated machinery required in modern continuous fat-splitting as compared with the very simple equipment used in the ordinary soap-boiling or the Twitchell process.

First Continuous Processes

One of the oldest patents for continuous fat-splitting was taken out by R. Tilghman in 1854[3] claiming "a process whereby oils and fats were hydrolysed by exposing them to water or aqueous alkali at high temperatures and pressures." Nothing apparently came of that patent. In 1922 Böhm[4] patented an apparatus which consisted essentially of a horizontal jacketed tube. Oil, previously emulsified, was pumped under pressure into the tube, while high-pressure steam was admitted into the outer jacket. A complicated system of valves, automatic pressure gauges, and by-passes was supposed to maintain the reaction at the desired temperature and pressure, but no commercial applications have been found for this invention. It was not until high-pressure equipment made in acid-resistant material became more popular that industry adopted continuous fat-splitting. The increasing demand for fatty acids of good quality and high purity in the last few years, too, prompted progressive large-scale manufacturers to adopt more modern processes.

Most of the successfully working continuous processes can be grouped under one of the following heads: (1) continuous spraying processes; (2) continuous processes using solid caustic soda; (3) continuous processes using water only. This is not a rigid division, but merely a convenient order based on certain common features of the various processes.

Continuous spraying processes are mainly employed where soap, and not free fatty acids, is the desired product, in addition to glycerine. The characteristic common feature is a spraying chamber. In the Lorenz process[5] the fats are thoroughly emulsified with the theoretical proportion of caustic soda or sodium carbonate, and then sprayed into a vacuum reaction chamber through which superheated steam is passing at a temperature above the melting point of anhydrous soap—say 250°-290° C. Under these conditions immediate saponification occurs and the particles of soap fall downwards in the reaction chamber, while the more volatile glycerol is carried off with the steam. This

process is well suited for the manufacture of soap powders.

A similar process was patented by Refining Inc.[6]. The oil or fat, mixed with caustic soda, is passed by a high-pressure pump into a reaction coil in which the temperature is maintained at 200° C. at a pressure of 250 lb./sq. in. Saponification is completed in about 30 seconds in a second reaction zone at 290° C. and 50 lb./sq. in. The mixture of soap, water, and glycerine is sprayed from the second coil into the separating chamber in such a way that the stream impinges against the walls and the soap moves down them in a thin stream. This chamber is kept under a vacuum of about 29 inches, and consequently the water and glycerol vaporise and can be subsequently condensed. Very pure sweet waters are obtained, containing no more than 0.1 per cent. soap, and the loss of glycerol in soap is negligible, because any that is not immediately volatilised in the vacuum chamber rapidly escapes from the soap as it flows in a thin film down the heated walls.

Continuous processes using solid caustic soda.—In this group only Kokatnur's process[7] is of interest. The oil or fat is dissolved in kerosene and saponified with anhydrous powdered soda. As saponification proceeds, the practically anhydrous glycerol is distilled off. The residual soap gel is washed to remove kerosene and glycerol. No industrial application of this process has been reported. J. Jacobs, however, recently described encouraging results from further experiments[8]; almost 90 per cent. sweet-water concentrations have been obtained and it is claimed that steam saving is about 90 per cent. Thus the glycerol could be recovered as dynamite glycerine without further evaporation.

Continuous processes using water only.—This is probably the most important group of continuous fat-splitting processes worked on a large scale with satisfactory results. No chemicals are added to the charge; but it is advisable to de-aerate both the oil and water before mixing, as any oxygen present may darken the colour of the fatty acids at the high working temperatures. The products are fatty acids which can be used for soap manufacture, and glycerine water of 30-35 per cent. concentration. Two types of these processes are known, one in which the charge and the water flow countercurrent in the reaction zone, and one in which they are in uniflow.

The Proctor and Gamble method is of the first type[9, 10]. Superheated steam is injected into the fat to be hydrolysed, which condenses under the prevailing working pressure of about 600 lb./sq. in. Some of the condensate dissolves in the fat while an excess remains free. No heat exchanger for the fat or oil is therefore required. This operation can be performed in a separate high-pressure vessel, but is preferably carried out in the lower part of the hydrolysing chamber itself. The hot oil/water mixture is then introduced into the bottom of the vertical reaction chamber where it meets a stream of water in countercurrent, which has been preheated to about 360° F. under a pressure sufficient to prevent volatilisation of water. The water percolates downward through the fat/water solution, thereby washing out from it any glycerine liberated by the fat-splitting. The sweet water is withdrawn from the bottom

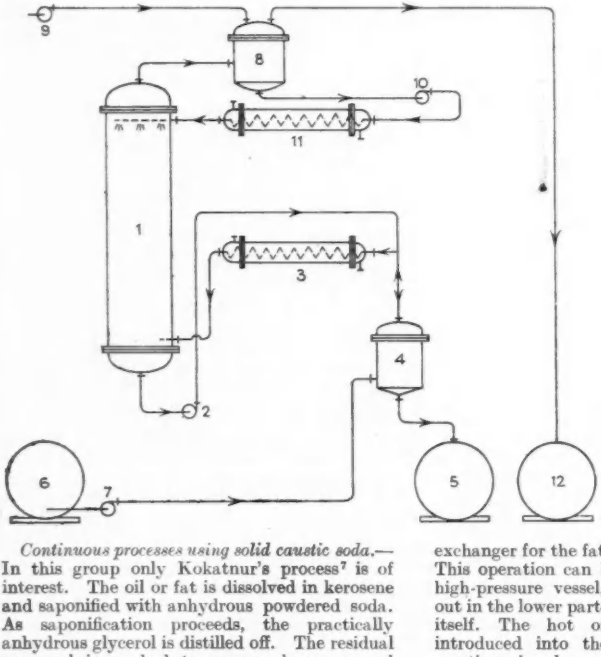


Fig. 2. Counter-current Fat-Splitting: the Colgate - Palmolive-Peet Process.

1. Reaction chamber.
2. Sweet water circulating pump.
3. Heater for sweet water and fatty oil mixture.
4. Sweet water separator.
5. Sweet water tank.
6. Fatty oil supply tank.
7. Fatty oil supply pump.
8. Free fatty acids separator.
9. Water supply pump.
10. Circulating pump.
11. Heater for water-fatty acids mixture.
12. Free fatty acids tank.

of the chamber, while the fatty acids ascend to the top from where they are withdrawn continually. The reaction chamber may be up to

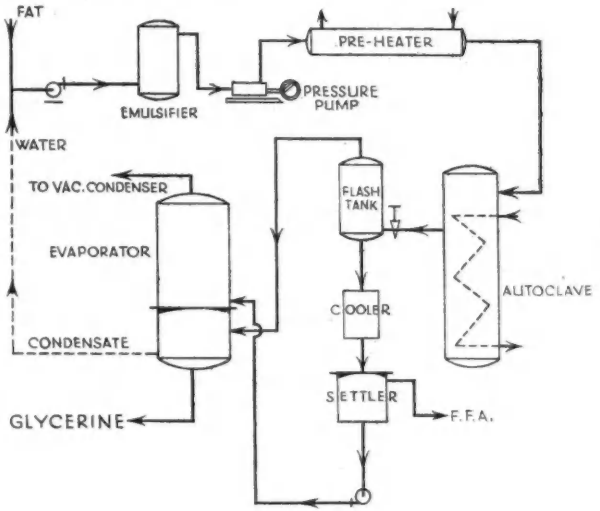


Fig. 3. Flow Diagram of Eisenlohr Uniflow Fat-Splitting Process.

50 ft. high; glycerine yields up to 98-99.5 per cent. can be obtained. The time of contact between oil and water is about 30 minutes. The sweet water withdrawn from the bottom of the hydrolyser goes to a reduced pressure zone where some of the water flashes off, giving a sweet water with a glycerine content of 35 per cent.

The process operated by the Colgate-Palmolive-Peet Co.[11,12] is similar to the one just described in so far as a mixture of oil and water is rapidly heated under pressure to the desired hydrolysing temperature and is then subjected to hydrolysis by countercurrent flow with hot liquid water. It differs in respect of the working pressures, which may be 200-250 lb./sq. in. in excess of the pressure of saturated steam at the temperature employed. Fat-splitting takes place in two stages.[1] Either two separate vessels may be used, or the first stage takes place in the lower part of a single column in which temperature and pressure conditions can be regulated independently from the second stage in the top of the column[13].

A simplified flow diagram of this process is reproduced in Fig. 2. Fatty oil is pumped from 6 to 4, where it meets hot sweet water. As the latter descends, it gives up some of its heat to the charge, while the latter percolates upwards and is pressed into heater 3 together with another portion of sweet water which is recirculated. The arrangement at the top of the column is similar. Water is pumped by pump 9 into vessel 8 where it meets free fatty acids in countercurrent and takes up some of their heat. A portion of the fatty acids is then recirculated with the fresh water by pump 10. A good control of temperature can be achieved by this operating scheme.

Of the processes in which the oil undergoing reaction and water are in uniflow the Eisenlohr and Hoffmann processes may be mentioned. In the former[14] the oil and fat are emulsified before entering the pre-heater (Fig. 3) and are then passed into the horizontal reaction chamber (of which there may be one or two), which consists of a narrow bore pipe and an outer tube. High pressure steam or Dowtherm is circulated in the annular space. Pressures inside the reaction tube are from 2500-3500 lb/sq. in., and the mixture of water, glycerine and fatty acids is released into a flash chamber in which a substantial part of the water evaporates, while the rest collects at the bottom of the chamber. The mixture is then passed through a cooler into settlers, where the sweet water (approx. 35 per cent. concentration) separates from the fatty acids. The sweet water is subsequently concentrated in a vacuum evaporator by the steam from the flash chamber. Great economy of steam is thereby achieved.

Hoffmann's[15] process is the older of the two. In this also oil and water are emulsified first, then heated and passed into a reaction tube, in which almost instantaneous hydrolysis is claimed at about 420-480° F. and about 900 lb./sq. in. The products of the reaction are also released into a flash chamber, but instead of entering settling tanks they are separated by fractional condensation. This process seems likely to give a glycerine which is high in fatty-acid impurity.

In some quarters anxiety has been expressed for the post-war market of the products of fat-splitting. It is argued that glycerine consumption especially will suffer inevitable and considerable reduction when the war ends. A timely reminder not to underestimate the great potential outlets for glycerine after the war has come from Mr. E. H. Little in the presidential address he gave to the Association of American Soap and Glycerine Producers in January. "There will be," he said, "a large post-war market for glycerine, as many applications of this product developed during the war will continue in peace time, and expanded research on new uses will go along with increased production." He also predicted greater interest in cleanliness due to military training, and therefore an increased demand for soaps of all descriptions. A good example how sometimes a new market is opened for a product in relatively abundant supply, is the consumption of 100,000,000 lb. of soap annually in the synthetic rubber programme of the U.S.A.

One of the outstanding uses of glycerine in recent years is in the field of synthetic resins and plasticisers. Manufacture of the alkyd resins used in the paint and varnish industry, consumes considerable quantities of glycerine. It serves also as one of the raw materials in the preparations of plasticisers of the type of Diacetin and Triacetin, and can be used successfully for the production of transparent plastics of the acrylate type.

Quite apart from their use in the soap, candle and allied industries, fatty acids have lately come into use for an increasing number and variety of purposes, and it is impossible to foretell what advanced industry employing modern methods of fractionation has in store for them. Already sebacic acid is used for making nylon and undecylenic acid for insecticides. Others are used in the plastics industry and as plasticisers and linoleic and linolenic acids derived from linseed oils are converted into a synthetic quick-drying oil, that takes the place of tung oil.

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Seven oil refineries in Hungary and Yugoslavia were attacked by a force of 500-750 American bombers on June 14. One of the targets hit was the Shell Koolaz refinery—the largest and most important in Hungary, five miles south of Budapest.

Institute of Fuel

Students' Medal

TO encourage the preparation of papers by students of fuel technology, the Council of the Institute of Fuel has decided to make an annual award of a medal, together with a prize consisting of books and/or instruments to the value of £5. The principal rules governing the award of this prize and medal are as follows.

1. The Council of the Institute of Fuel invites competition for a Medal, together with a prize of books or instruments to the value of £5 to be awarded annually for a paper submitted by a student member of the Institute or by a student under 25 years of age of any university or technical college in the United Kingdom.

2. The paper shall deal with some subject relating to the preparation or utilisation of fuel, or allied subjects. While the student is left free to choose his own subject if he so desires, the following subjects are suggested as a guide to his choice: (a) The industrial use of any two of the following gases: Town Gas; Coke-Oven Gas; Producer Gas; Blast-Furnace Gas; Water Gas; Natural Gas. (b) Trend of development in high or low temperature carbonisation of coal. (c) What is our present knowledge about the specific heat and heat conductivity of coal and coke? (d) Principles of screening. (e) Factors influencing the efficient blending of solid fuels. (f) Fundamental principles influencing the segregation of coal in bunkers. (g) Possible developments in the use of petroleum gases.

3. Papers must be submitted to the secretary of the Institute under a "nom de plume," the name and address of the author being enclosed in a sealed envelope and sent with the paper. Papers must be received by the secretary on or before September 1 in any year.

4. The name of the successful competitor in each year will be announced at the October meeting of the Institute, and the award presented at the annual dinner or luncheon, at which the prize winner will be the guest of the Institute.

5. Papers must be limited to a maximum of 6000 words, and may be illustrated by line drawings or photographs.

6. One medal and prize will be available for award each year.

7. The Institute reserves the right to withhold granting a medal in any year if, in the opinion of the Papers Committee, no applicant deserves the award.

8. In judging the papers submitted, consideration will be given to: (a) subject matter; (b) evidence of analytical power and logic; (c) construction of paper in so far as it gives evidence of an orderly mind; (d) English.

Parliamentary Topics

Materials for Export Trade

IN the House of Commons last week, Sir Stanley Reed asked the President of the Board of Trade whether, with the greater supply of raw material now available, preference would be given to the production of goods for export rather than for home consumption. The President of the Board of Trade, Mr. Dalton, replied that the principal factor now limiting the production of goods, both for home consumption and for export, was the supply of labour, on which, at this critical stage of the war, ever increasing demands were being made, and would continue to be made, in support of our military operations.

Sir S. Reed: May I ask my right hon. Friend if he does not agree that it is in the long-term interest of the community to encourage the export trade, even if it means going without goods we should like at home? —Mr. Dalton: Within reasonable limits, I think that is true, but it is possible to carry that policy too far. At the moment, we must give first preference neither to export nor to home consumption, but to supporting our fighting men across the water.

Chemical Welfare Research

Mr. Parker asked the Minister of Supply what steps he was taking to ensure that, in spite of the fact that chemical warfare had not so far been used by the enemy, the efficiency of the research services that had been established was maintained and the dispersal of the experienced personnel prevented. Mr. Sandys assured the questioner that research in the field of chemical warfare was being energetically pursued and a powerful team of scientists, technicians and Service personnel is engaged upon this work.

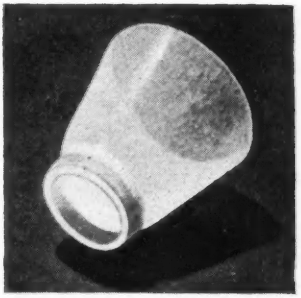
CHEMICAL STONEWARE PUMP

About two years ago the Pneu-Pump appeared on the market as a compact, self-contained, compressed-air-operated pump having no moving parts and being specially designed for the pumping of liquids containing solid matters in suspension. Since its introduction this pump has proved eminently satisfactory in many widely differing branches of industry: sewage, slurries, oils, chemicals, viscous fluids, paints, hot liquids, clinker-laden water—all these are, to-day, being handled by the Pneu-Pump. Recent advances have been made which still further increase the usefulness of this pump. One of these is the perfecting of a means whereby steam can be made to take the place of compressed air in operating the pump. This is done by taking a valve-controlled connection from the steam range and passing the steam through a jet blower. A quantity of air is thus entrained and compressed. The mixed steam and air then pass to the pump which operates in the usual manner.

Another development of great importance to the chemical industry, is the production of a model made of chemical stoneware for the pumping of acids or other highly corrosive liquids. This design can be conveniently coupled to standard stoneware pipes and, as the separate parts are fastened together by external clamps, no metallic parts can come into contact with the liquid that is being pumped. Full details are given by the makers, Ames Crosta Mills & Co., Ltd., Heywood, Lancs., in a recently published booklet.

FILTERING CRUCIBLES

The Thermal Syndicate, Ltd., Wallsend, Northumberland, advise that they are now able to supply Vitreosil crucibles for filtration with subsequent ignition at temperatures up to 1000°C. The need for preparing asbestos filtering mats required for the ordinary Gooch crucibles is thus obviated. Filtration, ignition, and weighing are all



done in the same crucible, affording an enormous saving of time and conducive to greater accuracy. Laboratory workers will appreciate the value of these advantages of the Vitreosil filtering crucible. The established grades of porosity of porous glass filtering crucibles, Nos. 1, 2, 3 and 4 (the last being the finest) have been adhered to and apparatus can be supplied incorporating discs of any of these grades. A descriptive leaflet with prices will be sent on application to the manufacturers.

A deposit containing 5.9 per cent. cobalt has been found about 20 km. west of Panes (Oviedo), Spain. In spite of the arsenic present, a satisfactory extraction of cobalt has been achieved, and production is expected to reach 200 tons a month.

Chemical Industry in Italy

Montecatini's Annual Report

THE recently published annual report of the Montecatini combine of Milan gives an interesting picture of the development of Italy's chemical industry in the momentous year 1943. Financial results, to begin with, show a marked improvement over 1942. Gross profit increased from 471.8 million to 688.5 million lire; net profit is shown at 216.7 million against 179.2 million lire a year ago. A net dividend of 9 per cent. was declared on the capital, which was increased from 1600 million lire to 2000 million lire in 1942.

Plant, equipment, machinery, etc., leaped from 1370.1 million lire to no less than 2828.3 million lire owing to the absorption of the "Ammonia e Derivati" (Soc. Gen. per l'Industria dei Prodotti Azotati Sintetici) as well as of the Soc. Elettrica Alto Adige. Stocks of raw materials and finished products stand at 423.3 million, compared with 270.8 million lire in 1942. The balance sheet reveals that the company owns 270.8 million State issues and 948 million lire other investments, compared with 128.3 and 954.5 million lire, respectively, a year ago. As a result of the inflation of currency cash rose from 16.7 million to 56.2 million lire: reserves increased from 1097.5 million to 1313.6 million, while debtors now total 2230.3 against 1591.6 million lire. Depreciation account shows an increase of over 1000 million lire, having risen from 934.2 million to 1942.7 million lire in the year under review. General expenditure is 89.6 million, compared with 66.6 million.

Effects of the War

The report reveals that 18 of the group's 200 plants are in that part of Italy which is under Allied occupation. The value of these is stated to be over 500 million lire. The report maintains a discreet silence concerning plant destroyed or damaged by military operations or sabotage in the German-held part of the country. The cessation or interruption of work in many plants, and the large-scale and very expensive decentralisation measures combined with an increase in the cost of materials and labour, are stated to have brought about a substantial increase in the cost of production. Although having to face unprecedented difficulties, the Board nevertheless expresses the hope that the combine will have an important part to play in the country's future.

As regards the group's manifold interests, production of coal, sulphur and pyrites was on a satisfactory level in the first half of 1943; later, however, a marked decline of output was noticeable. The marble-quarrying industry was fully occupied and, in view of the likelihood of a substantial post-war building activity, prospects are satisfactory. An important subsidiary, the I.N.A. (Industria Nazionale Alluminio) maintained its production in spite of the loss of the bauxite mines of Manfredonia. Production of light metal alloys, on the other hand, controlled by the Soc. Lavorazioni Leghe Leggere, showed a decline. The war brought about a further decline in the output of phosphate fertilisers; production of synthetic nitrates, however, was well-maintained and there was a slight increase in the output of lime fertilisers. The scarcity of copper had serious effects on the production of insecticides. A number of new brands containing very little or no copper at all, were marketed (as has been recorded in our columns).

Decline in Production

As regards heavy industrial chemicals, the report merely states that after an intensification of production in the first half of the year, output declined by about 30 per cent. in the second half. Technical progress was made in the production of resins and varnishes. The rayon plant "Rhodiaceta Italiana," using the patents of the French concern, Comptoir des Textiles Artificiels Usines Rhône Poulenc, had to cut down production owing to raw material difficulties. On the production of synthetic fuels, the report states that the management has "lost contact" with the A.N.I.C. (Azienda Nazionale Idrogenazione Combustibili) operating a hydrogenation plant at Bari, while the company's large plants at Leghorn, damaged in May by allied bombers, ceased production in September. According to another reference, the Leghorn plant was dismantled and removed to an undisclosed locality. There was also a serious decline in the output of the A.C.N.A. (Azienda Colori Nazionali ed Affini), the dyestuff subsidiary, 49 per cent. of whose share capital is in the hands of I.G. The Soc. Farmaceutici Italia of Settimo Torinese registered an increase in both output and demand. This subsidiary also co-operates with the Rhône-Poulenc group. A number of new products were marketed by this company, including a morphine substitute, called "Metidina," and a synthetic drug anti-malarial called "Sele."

Production of explosives and munitions by the Soc. Gen. Esplosivi e Munizioni was more or less normal for the first eight months; later in the year, however, the plant was seriously damaged by bombing and part of it had to be evacuated. As regards Montecatini's mining interests, the board was cut off from the lead mines and smelters of Sardinia.

The War Production Board of the United States has surplus stocks of mercuric chloride amounting to 364,450 lb., representing a normal supply for 5 months based on the 1943 rate of sales.

Personal Notes

MR. E. OSWALD TOFT has been appointed chairman of British Alkaloids, Ltd., in succession to the late Dr. E. J. Parry.

MR. DAVID V. EVANS, chemist at Baldwin's, Ltd., Landore, Swansea, has received his R.A.F. wings and commission in Canada.

MAJOR D. A. POND, of the 5th St. Austell Battalion, Home Guard, was awarded the M.B.E. in the recent Birthday Honours. For the past 15 years Major Pond has been chief engineer to the firm of Parkyn & Peters, china clay producers, St. Austell, Cornwall.

The following officers were elected at the annual meeting of the Birmingham and Midland section of the Society of Chemical Industry last month. *Chairman*, MR. R. S. POTTER; *vice-chairman*, MR. C. DIAMOND; *hon. secretary*, MR. J. W. CRUMP; *hon. treasurer*, MR. W. T. COLLIS; *hon. auditor*, DR. S. R. CARTER.

The annual meeting of the Industrial Pest Control Association elected the following officers for 1944/1945: *President*, DR. E. HOLMES (Plant Protection, Ltd.); *vice-president*, MR. A. F. MCINTOSH (Thomas Harley, Ltd.); *hon. treasurer*, MR. S. F. SPRANGE (London Fumigation Co., Ltd.); *executive committee*, MESSRS. F. H. BRAYBROOK (Technical Products, Ltd.); C. S. KREGOR (W. Edmonds & Co., Ltd.); E. MCLAREN (Haller Laboratories, Ltd.); T. NAPIER (I.C.I., Ltd.); J. E. R. SIMONS (Geo. Monro Ltd.); J. B. WILTON (Fumigation Services, Ltd.). MR. A. J. HOLDEN, B.Sc., F.I.C., and MR. W. A. WILLIAMS, B.Sc., of the A.B.C.M., are undertaking the secretarial duties.

Among the new foreign Fellows of the Royal Society who were elected last week is DR. THEODOR SVEDBERG, professor of physical chemistry at the University of Upsala, Sweden, since 1912. He is especially celebrated for his work on colloid chemistry, for which he was awarded a Nobel Prize in 1926. Professor Svedberg's name is associated with the introduction of the ultracentrifuge, a device whereby the molecular weights and size distribution of sol particles may be determined. Recently he has been engaged in efforts to obtain synthetic rubber from native Swedish materials, and a polysulphide has been produced, called "Modotiol A," which is used to replace rubber for certain uses (*see* THE CHEMICAL AGE, November 21, 1942, p. 450).

DR. ALEXANDER FLECK, recently appointed a director of I.C.I., graduated in 1911 as B.Sc., at Glasgow University. Thereafter he was employed as physical chemist to the Glasgow and West of Scotland Radium Committee for research on cancer. In 1916 he was granted the D.Sc. degree for his research work on the chemistry of radioactive elements. In the following year he became chemist to the Castner-Kellner Alkali Co., Wallsend, and in 1921 was appointed a member of the executive committee, Cassel Cyanide Co., Glasgow. In 1924, Dr. Fleck went to the Runcorn works of the Castner-Kellner Alkali Co., and, following the formation of I.C.I. in 1927, was in 1928 appointed alkali division manager, and in 1929 delegate director of Synthetic Ammonia and Nitrates, Ltd. He became managing director of the General Chemical Group of I.C.I. in 1931, and in 1937 was appointed chairman of the company's Fertiliser and Synthetic Products Group.

Dr. A. Fleck.

Obituary

SIR PRAFULLA CHANDRA RAY, C.I.E., D.Sc., Ph.D., emeritus professor of chemistry at Calcutta University, died on June 16, aged 83. Educated at the Presidency College, Calcutta, and Edinburgh University (where he was vice-president of the chemical society), he returned to India in 1888 and for 25 years held the appointment of Professor of Chemistry in his old college. Later he was Professor of Chemistry of Calcutta University until 1937. He was throughout his career a leading figure in the advancement of chemical education in India. He actively encouraged the establishment of a College of Science in Calcutta, and founded the Indian School of Chemistry and the Indian Chemical Society, of which he was the first president (1924-26). In 1922 he renounced his professorial salary to provide a fund for the furtherance of chemical research. By no means purely academic, he interested himself in the economic development of Bengal, and served as founder-director of the Bengal Chemical and Pharmaceutical Works. The most striking result of his numerous chemical researches was the discovery, in 1896, of mercurous nitrate.

General News——From Week to Week

Cocoa butter may now be supplied to trade users for pharmaceutical purposes without a buying permit, in accordance with the Edible Oils & Fats (Control of Sales) Order, 1944 (S.R. & O. 1944, No. 672).

Specification DTD 646, for high-tensile aluminium alloy sheets and coils (solution-treated and artificially aged) has been reprinted, incorporating Amendment List No. 1, and is available from H.M.S.O., price 1s.

A simple piece of apparatus, designed for studying the sorption of fumigant vapours by stored food products under fumigation conditions, is described by F. P. W. Winteringham in *J.S.C.I.*, May, 1944, p. 144. The specific fumigant studied was ethylene dichloride.

A Human Nutrition Research Unit has been established in London by the Medical Research Council, with Dr. R. S. Platt as director. It will be specially occupied in the improvement of the health of British Colonial peoples, and will co-operate closely with workers in the Colonies.

The Minister of Production stated in Parliament last week that he was taking steps to facilitate the substitution of removable for permanent black-out in factories throughout the country. He was informed that removable black-out was already installed in 95 per cent. of factories in Lancashire.

The British Plastics Federation has set up a committee to advise St. Dunstan's on the training and placing of blind trainees in the plastic-moulding industry. The June number of the Ministry of Labour's Production and Engineering Bulletin tells the story of a blinded soldier who is now operating a nine-impression die for making plastic hand-grenade parts.

Only small increases are noticeable in the wholesale prices of chemicals and metals for May. The Board of Trade index figures for coal and for chemicals and oils remained stationary at 201.5 and 151.6 respectively (1930 = 100), while those for iron and steel and for non-ferrous metals each had an increase of 0.1 per cent., respectively from 183.0 to 183.2, and from 127.4 to 127.6.

Foreign News

A local section of the American Chemical Society has organised a speaker's panel of 40 members who are prepared to lecture to civic clubs and Servicemen.

Sulphuric acid production in Belgium is now concentrated in 8 plants, it is reported. Before the war the country made 800,000 tons a year.

The Zagyvafia Glass Factory in Hungary, with a capital of 2,500,000 pengö, has opened a new plant for the production of glass wool, silk and textiles. Splinter-proof glass will also be made there.

Benzene sulphonamide, known for nearly a century, but still without any large-scale use, is being made by the Wyandotte Chemicals Corporation of Michigan in two grades, 85-95 per cent. and 98-100 per cent. pure.

The Hercules Powder Company of America is marketing a new compound called Stabinol for stabilising unsurfaced dirt roads. It is claimed that where traffic is light such roads can be treated at a cost of 30 cents a square yard. Already widely used by the U.S. Army, Stabinol is superseding the pine-rosin chemical called Vinsol, introduced earlier for this purpose.

A new technical research institute, established in co-operation with the Higher Council for Scientific Research (which granted an initial subsidiary of 125,000 pesetas) has been opened by the provincial authorities of Barcelona. The new institute is concerned with the more intensive utilisation of domestic raw materials and with advising the establishment of new industries or the modernisation of existing ones.

Shortage of artificial fertilisers in India has led the Food-Grains Policy Committee to review the question of ammonium sulphate supplies. In 1939, consumption was 96,000 tons, of which only 30,000 tons could be met by domestic production. The committee estimates that 350,000 tons could be added to present capacity, but any solution of the problem is likely to be deferred owing to the difficulty of importing equipment.

The photo-reduction of ferric chloride in the presence of acetone and ether has been investigated by Mata Prasad and P. R. Bavdekar, of the Royal Institute of Science, Bombay (*Proc. Ind. Acad. Sci.*, Section A, 1943, *18*, 6, p. 373). They find it to be a unimolecular reaction, the velocity content of which is affected by temperature and the frequency of the exciting radiations. The rate of reaction is directly proportional to the intensity of the incident light.

A new link between university and industry has been forged in America with the grant by Westinghouse of $75,000 for a 5-year training and research project at Purdue University for the "intensive training of graduate students in exploring the field of heat transfer." This project will come under the direction of Dr. G. A. Hawkins, of the Purdue faculty, and Dr. M. Jakob, of the Illinois Institute of Technology.

Rumania is extending the cultivation of oil-plants especially for the production of castor oil on an area of 10,000 hectares.

Synthetic rubber production in the United States and Canada during May reached an all-time peak of almost 70,000 tons.

A new deposit of fullers earth has been discovered near Oudjda, in north-eastern Morocco.

The synthetic oil centre of Gelsenkirchen was bombed for the third time in less than three weeks when a force of Mosquitos attacked it on June 14.

A Reuter message from Washington states that the U.S. House of Representatives has rejected an amendment to the Price Control Bill, which would have increased the crude oil price about 35 cents a barrel.

Holzverzuckerungs A.G., Graubünden, Switzerland, has enlarged its plants to a monthly production of more than 500,000 litres, 100 per cent. alcohol and the corresponding production of yeast.

Swedish home production of aluminium has made her already independent of imports and has reached a capacity of more than 5,000 tons per nnum. Home-mined andalusite and cryolite are the raw material, and the two main works are at Mansbö and Kubikenborg.

The campaign against the enemy's oil resources continues unabated. On Friday last week Bomber Command attacked the synthetic oil centre of Sterkrade, north of Duisburg, while throughout the week refineries in the Balkans and Austria were bombed.

Refined paraffin wax can be produced against darkening on prolonged exposure to light by the admixture of a small amount of triamyl-, tributyl-, diamyl- dibutyl-, or monobutyl-amine. This claim is made in U.S.P. 2,331,469 by the Socony Vacuum Oil Co.

No major extensions of existing penicillin plants in the United States will now be approved, states *Chem. Met. Eng.* The Chemicals Bureau of the War Production Board is credited with having said that only minor adjustments necessary for eliminating production bottlenecks will be allowed, although some consideration will be given to firms developing new processes of making the drug.

Copper ammonium fluoride—with the approximate formula $Cu(NH_3)_4F_2$—is being produced by the Pennsylvania Salt Manufacturing Co., Philadelphia, in trial quantities. In aqueous solution, it is effective for mildew-proofing textiles. The same firm is making an 86 per cent. pure aluminium fluoride, which is finding use as a flux in aluminium manufacture and in the making of white ceramic enamels.

A new company has been formed in Hungary with a capital of 200,000 pengö to undertake vitamin research and production. Professor Szentgyörgyi is collaborating in the new venture, states a Swiss journal, and one of the projects contemplated is the large-scale cultivation of paprika, a rich source of vitamin C.

An antimony-titanium opacifier, with the trade name of "Uverite" and an approximate formula of $7CaO.CaF_2.6TiO_2.2Sb_2O_5$, has lately been put on the American market for use instead of tin oxide in enamel manufacture. It is claimed to be a sound substitute in so far as its effect on opacity, colour, gloss, firing range, general workability and durability is concerned.

A report on the quicksilver deposits in the Cinnabar Creek area, in the Georgetown and Akiak districts of S.W. Alaska, has been prepared by the U.S. Geological Survey. The quicksilver districts are little explored and poorly exposed; the type and extent of mineralisation are similar to those at the better known deposits at Sleitmut and De Courcy Mountain.

Alcohol from wood waste is to be made in a new $2,247,000 plant at Springfield, Oregon, belonging to the Willamette Valley Wood Chemical Co. The annual capacity is estimated at over 4,000,000 gallons. Production of 7,000,000 gallons a year is expected from a unit built for the United States Sugar Corporation at Clewiston, Florida; the raw material there will be sweet potatoes of high starch-content.

Hoarding of drugs and pharmaceutical products of all kinds is reported from Germany, and the authorities have issued an appeal to the public not to aggravate what is officially described as "a senseless run" on these supplies. According to the head of the German pharmacists' association, production of pharmaceutical preparations has trebled during the war, and though the larger part goes to the Forces, pharmaceuticals continue to be one of the few classes of goods that are unrationed.

Forthcoming Events

A meeting of the **Electrodepositors' Technical Society** takes place at the Northampton Polytechnic, St. John Street, London, E.C.1, on **June 26,** when a paper on "The Estimation of Ammonia in Electrolysed Cyanide Solutions" will be read by Messrs. C. M. Blow, N. G. Hiscox, and M. W. Smith. Two films, illustrating "The Manufacture of Accumulators" and "Electrodeposition" will follow.

Dr. I. Broda will deliver a lecture entitled "Prominent Austrian Scientists," at the

next meeting of the **Association of Austrian Engineers, Chemists and Scientific Workers,** arranged for 7.30 p.m., on **July 3,** at the Austrian Centre, 69 Eton Avenue, Swiss Cottage, London, N.W.3.

The London Section of the **Oil and Colour Chemists' Association** has arranged a series of post-graduate lectures, to be given by Professor E. K. Rideal, F.R.S., on the subject of "Colloid Science—Surface Action." The lectures take place at 6.30 p.m., on **July 6, 20** and **27,** respectively at the London School of Hygiene and Tropical Medicine, Keppel Street, W.C.1. The inclusive charge for the three lectures is 10s., to be paid in advance. Junior members are invited free of charge, but should inform the hon. secretary.

Arrangements have been made to hold the annual meeting of the **British Pharmaceutical Conference** at the Waldorf Hotel, Aldwych, London, W.C.2, on **July 11.** There will be three science sessions. A light lunch and a lounge tea will be provided and an inclusive day ticket at 10s. 6d. may be obtained by making application to the Hon. Secretaries, British Pharmaceutical Conference, 17 Bloomsbury Square, London, W.C.1, not later than June 27.

The London Section of the **Society of Chemical Industry** holds a joint meeting with the **Institute of Metals** on **July 13,** at 7.30 p.m., in the hall of the Institution of Mechanical Engineers, Storey's Gate, Westminster. Mr. A. R. Powell, of Johnson Matthey and Co., Ltd., will read a paper on "The Minor Metals."

The annual general meeting of the **Institute of Factory Managers** will be held at the Hotel Russell, Russell Square, London, W.C.1, at 3 p.m., on **July 15.**

Company News

Reckitt & Sons, Ltd., report a profit for 1943 of £458,099 (£460,194), and are repeating the 22½ per cent. ordinary dividend.

Gloucester Manganese Mines, Ltd., report a dividend for the year of 2½ per cent. (14 per cent.).

South African Torbanite Mining & Refining Co., Ltd., with a final payment of 5 per cent., are distributing an ordinary dividend of 10 per cent. (nil) for the current year.

The Fullers' Earth Union, Ltd., reports a net profit of £22,483 (£20,789), for the year ended March 31. The unchanged 15 per cent. dividend has already been recorded.

Cooper, McDougall & Robertson, Ltd., report a dividend of 6¼ per cent. for the 15 months ended December 31 (5 per cent. for 12 months). Net profit was £90,550 (£87,266).

Boots Pure Drug Co., Ltd., are paying a final ordinary dividend of 10 per cent., making a total of 30 per cent. (24 per cent.). Net profit for the year ended March 31, after taxation, was £533,834 (£434,970).

Goodlass Wall and Lead Industries, Ltd., record for 1943 a net profit of £171,621 (£121,972). The dividend, as already announced, is 7 per cent. (6 per cent. and 1 per cent. bonus).

Thomas De La Rue & Co., Ltd., announce a final dividend of 30 per cent. for the year ended March 31, making 40 per cent. This compares with a dividend of 35 per cent. for the previous 15 months, equivalent to 28 per cent. per annum.

Fisons, Ltd., announce a final ordinary dividend of 7½ per cent., making 10 per cent. for the year ended September 30 (12½ per cent. for 15 months). An interim dividend of 2½ per cent. for the current year is also announced.

The final dividends of the **Cape Asbestos Co., Ltd.,** for the year to April 12, are 12½ per cent. on both the ordinary and the preference shares, making a total of 15 per cent. (12½ per cent.) on ordinary, and 20 per cent. (17½ per cent.) on preference. Profits for the year were £115,250, against £169,209, but taxation accounts for only £47,550 compared with £111,500.

Commercial Intelligence

The following are taken from printed reports, but we cannot be responsible for errors that may occur.

Mortgages and Charges

(Note.—The Companies Consolidation Act of 1908 provides that every Mortgage or Charge, as described therein, shall be registered within 21 days after its creation, otherwise it shall be void against the liquidator and any creditor. The Act also provides that every company shall, in making its Annual Summary, specify the total amount of debt due from the company in respect of all Mortgages or Charges. The following Mortgages and Charges have been so registered. In each case the total debt, as specified in the last available Annual Summary, is also given—marked with an *—followed by the date of the Summary, but such total may have been reduced.)

PREMIER BITUMEN AND ASPHALTE CO., LTD., London, E. (M., 24/6/44.) June 2, £7500 debenture, to C. R. de Berry, Woodford Green; general charge. *Nil. March 1, 1943.

Satisfaction

BRITISH CELANESE, LTD., Torquay. (M.S., 24/6/44.) Satisfaction June 1, of debenture stock registered March 5, 1942, to the extent of £400,000 (outstanding balance).

Chemical and Allied Stocks and Shares

BUSINESS in stock markets has been on a smaller scale than of late, although the tone generally remained firm and industrial shares held all but a small part of their recent substantial gains. British

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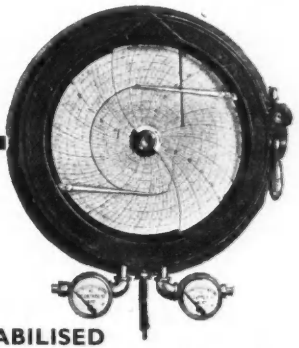

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We are the largest makers in the Country of Laboratory, Scientific and Technical Porcelain. . . We are making a supreme effort to maintain adequate supplies to meet the ever-growing needs of the Laboratories and Research Stations of war-time Britain. . . Supplies can be obtained by all registered Laboratory Furnishers.

THE WORCESTER ROYAL PORCELAIN CO., LTD., WORCESTER.

MAKERS OF THE BEST LABORATORY PORCELAIN

Funds continued very steady, reflecting a tendency for business to centre on high-grade investment stocks. Imperial Chemical at 40s. 4½d. were little changed on balance, and Borax Consolidated deferred were 37s., while at 47s. Dunlop Rubber held nearly all their recent further rise. Boots Drug were a good feature, rising from 48s. 9d. to 49s. 6d. xd. on the unexpected increase from 24 per cent. to 30 per cent. in the dividend, and the higher earnings which reflect the bringing in of larger profits from the subsidiaries. Lever & Unilever were also a good feature with a further rise to 38s. 6d., the view having gained ground that after the war there seem good prospects of dividends returning to the 10 per cent. level. Lever N.V. were 37s.

De La Rue at 188s. 9d. failed to hold all an earlier rise, although the increased dividend created an excellent impression. Among shares of other companies connected with plastics, British Industrial Plastics 2s. shares were 7s. 4½d., Erinoid again 11s., while elsewhere Lewis Berger moved higher at 105s. In other directions, following their recent advance, British Plaster Board 5s. ordinary showed a partial reaction to 34s. 9d., as did Triplex Glass at 42s. 9d. The market is continuing to talk of the possibility of a higher dividend on the last-named, although the recent strong rise in price mainly reflects encouraging views as to the post-war outlook.

Imperial Smelting at 14s. 6d. were firmer on attention drawn to the good yield of nearly 5⅝ per cent. on the dividend, which is generally expected to be maintained at 4 per cent. General Refractories 10s. ordinary at 18s. 3d. showed further improvement on the assumption that the upward trend in earnings is continuing, with scope for rising dividends after the war. In common with most of the recently active shares, British Celanese moved back slightly to 31s. 3d., and Courtaulds to 55s. 3d., but selling was on a moderate scale. Bradford Dyers were 24s. 4½d., Calico Printers 16s. 6d., but Bleachers improved slightly to 11s. 3d. Barry & Staines have been fairly steady around 50s., awaiting the dividend announcement. Nairn & Greenwich were firm at 78s. 9d., and Wall Paper Manufacturers deferred little changed at 45s. United Molasses were 34s. 6d., and at 97s. the units of the Distillers Co. were little changed, pending declaration of the dividend. Associated Cement have been steady at 66s.

B. Laporte showed dealings up to 83s. 9d. Ruston & Hornsby were slightly higher at 50s. xd., Staveley steady at 53s., Stewarts & Lloyds 56s. 7½d., Tube Investments 97s. 6d., Dorman Long 28s., and United Steel 27s. 3d. Neepsend Steel further strengthened to 31s. 6d., and Guest Keen to 38s. 3d. Consett Iron 6s. 8d. units were 9s. 4½d., and Richard Thomas 11s. In other directions, W. J. Bush were 63s. 9d., Greeff-Chemicals 5s. ordinary 8s., and Monsanto Chemicals 5½ per cent. preference 23s. 6d. Dealings around 26s. 3d. were shown in British Drug Houses, and up to 9s. 1½d. in British Glues & Chemicals 4s. ordinary. Morgan Crucible first preference have marked 27s. 3d. and the second preference 24s. 10½d. Valor ordinary were dealt in at slightly over 50s., while elsewhere, Fisons ordinary were 52s., following the dividend announcement. Gas Light & Coke were 21s. 3d., and Low Temperature Carbonisation 2s. ordinary 2s. 9d. Firmness at 27s. 1½d. was shown in Sangers ordinary, and Timothy Whites were 36s. British Oxygen were 83s. 3d., and British Aluminium 47s. 6d. Oil shares, following a general advance, showed a partial reaction, "Shell" being 82s. 6d., Burmah Oil 88s. 1½d. and Anglo-Iranian 122s. 6d.

British Chemical Prices

Market Reports

INQUIRY on the London general chemicals market during the week has covered a fairly wide range of materials, and the price position generally remains on a firm basis. Deliveries against existing contracts are reported to be satisfactory and replacement orders are coming through steadily. In the soda products section, acetate and nitrate of soda are a good market and a steady demand is reported for supplies of hyposulphite of soda. A moderate business is reported in Glauber salt and salt cake, and industrial refined nitrate of soda is meeting with a steady inquiry. Any offers of yellow prussiate of soda are quickly absorbed. A firm price position is reported from the potash section, with acid phosphate of potash in good demand. A steady inquiry is in circulation for both the pharmaceutical and technical grades of permanganate of potash, and supplies of bichromate of potash are being distributed to priority users. High prices are indicated for offers of yellow prussiate of potash and a steady demand is reported for acid phosphate of potash. In other directions glycerine is a good market, and there is strong demand for white powdered arsenic. Supplies of peroxide of hydrogen are quickly taken up and a fair trade is passing in alum lump. There is little to report on the coal-tar products market this week and a fairly steady business is passing in pitch for home trade requirements. The anthracene oils are scarce and a steady demand is reported for cresylic acid and carbolic acid. The benzols and toluols are in good request.

MANCHESTER.—Quotations on the Manchester chemical market during the past week have been firm throughout the range.

though little actual movement falls to be recorded. Deliveries of the general run of sodium compounds have continued on steady lines, and there has been fairly brisk movement of supplies of sulphate of alumina, formaldehyde, carbonate of magnesia, and the barium products, with most of the acids finding a ready outlet. Activity in the tar products section is mainly concerned with delivery against existing commitments.

GLASGOW.—In the Scottish heavy chemical trade conditions have been rather quieter during the past week for home business. Export trade remains rather limited. Prices keep very firm, with no actual changes to report.

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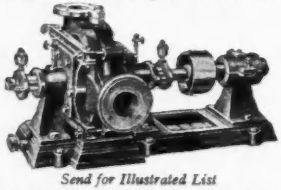

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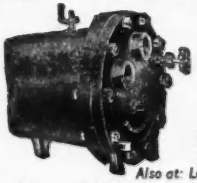

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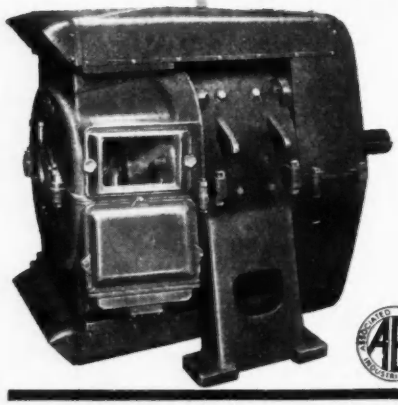

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